

Revision Log

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Operation of the Spectrace 9000 Field-Portable X-Ray Fluorescence Instrument

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Operation of the Spectrace 9000 Field-Portable X-Ray Fluorescence Instrument

1.0 PURPOSE

This standard operating procedure (SOP) describes the process for operating and using the Spectrace 9000 field-portable x-ray fluorescence (XRF) analyzer to screen for hazardous, or potentially hazardous, inorganic materials at the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship, Remediation Services (RRES-RS) project. The data that are generated allow for rapid evaluation of the extent of contamination. Samples are analyzed for elements of atomic number 13 (aluminum) through 92 (uranium), with proper x-ray source selection and instrument calibration. Environmental applications include measuring elemental metals in soils and on filters and measuring lead in paint. The success of this process directly ties to the participation of each employee within LANL, RRES-RS project.

2.0 SCOPE

- 2.1 All **RRES-RS project participants** shall implement this mandatory SOP.
- 2.2 **Subcontractors** performing work under the RRES-RS project's quality program shall follow this SOP.

OR

- 2.3 **Subcontractors** may use the subcontractor's procedure as long as the substitute meets the requirements prescribed by the RRES-RS Quality Management Plan, and the RRES-RS Quality Program Project Leader (QPPL) and a RRES-RS technical staff person approve the procedure before the subcontractor begins the designated activity.

3.0 TRAINING

- 3.1 **RRES-RS project participants** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.
- 3.2 **RRES-RS project participants** using this SOP shall document training in accordance with QP-2.2.
- 3.3 The responsible **project team leader (PTL)** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.
- 3.4 **RRES-RS project participants** may request any needed assistance with implementation of this procedure from RRES-RS Quality Integration and Improvement (QII).

4.0 DEFINITIONS

- 4.1 *Site-specific health and safety plan (SSHASP)*—Health and safety plan that is specific to a site or RRES-RS-related field activity that has been approved by an RRES-RS health and safety representative. This document contains information specific to the project including scope of work, relevant history, descriptions of hazards by activity associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment [PPE]) and hazard mitigation.
- 4.2 *X-ray fluorescence spectroscopy*—A nondestructive qualitative and quantitative analytical technique used to determine the elemental composition of solid, liquid, thin film, and powder samples.

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- Author
- Field team leader
- Operator
- PTL
- QPPL
- RRES-RS project participants
- User

6.0 BACKGROUND AND PRECAUTIONS

- 6.1 **RRES-RS project participants** shall use this SOP in conjunction with an approved SSHASP.
- 6.2 Background
- 6.2.1 The Spectrace 9000, energy-dispersive field-portable XRF instrument includes three compact, sealed-radioactive sources: iron (⁵⁵Fe), cadmium (¹⁰⁹Cd), and americium (²⁴¹Am), which are used for identification and quantization of 26 elements. The instrument is equipped with a high-resolution HgI₂ detector connected to a sealed electronic module.
- 6.2.2 The electronic unit contains a 2048 multi-channel analyzer for spectral analysis. It provides internal nonvolatile memory for storage of 120 spectra and results for 300 multi-element analyses, which are shown on the instrument's display panel. An RS-232C serial port allows users to download data and spectra to

a peripheral device such as a printer or IBM-compatible personal computer.

6.2.3 A 115-/220-V wall outlet or a four-hour, rechargeable battery powers the Spectrace 9000. The device operates in temperatures ranging from 32°F to 120°F. Furthermore, the probe and electronic unit may sustain exposure to a light rain. The enclosure of the system (electronic unit and probe) in the optional, water-repellent carrying case provides additional protection. Rubber gaskets seal the probe and electronic unit that with soap and water decontaminates.

6.2.4 The probe battery requires replacement once every six months. This requires the opening the top of the probe. Otherwise, the probe should remain closed and covered with the safety cover, laboratory safety cover, or laboratory safety shield when not in use.

6.3 Precautions

6.3.1 Instrument integrity—Immediately notify the Spectrace instrument manufacturer of any condition or concern relative to the probe's structural integrity, source shielding, source switching condition, or operability. Immediately notify the appropriate State agency or the US Nuclear Regulatory Commission office of any damage to the radioactive source or of any loss or theft of the device.

6.3.2 Labels and certifications—Do not alter or remove the labels or instructions on the probe(s). Leak test the source(s) in the probe every six months, and ensure that the leak-test certificates accompany the instrument at all times.

6.3.3 Handling—Do not drop or expose the Spectrace 9000 to conditions that produce excessive shock or vibration. Never pull the probe cable while unplugging the probe. Grasp the probe plug at the ribbed metal connector, squeeze, and pull gently while the connector is unplugged. Never forcibly plug in the connector. Only rotate the handle of the electronic unit when the release buttons on the side of the handle are depressed. Do not store the Spectrace 9000 at an ambient temperature below -4°F or above 110°F.

6.3.4 Transport—Properly package the Spectrace 9000 during transport. The suitcase that accompanies the unit is acceptable. DOT requires that the following statement accompany the unit during transportation: "This package conforms to the conditions

and limitations specified in 49 CFR 173.422 for acceptable radioactive material, instruments, and articles, UN 2910.”

7.0 EQUIPMENT

The Spectrace 9000 XRF instrument includes the equipment listed below.

- Analyzer unit for data acquisition, processing, and display
- Hand-held probe
- Three pure element sources (^{55}Fe , ^{109}Cd , ^{241}Am) and a safety cover
- Probe laboratory base stand with safety shield
- Interconnecting cable
- RS-232C serial I/O interface cable and connector
- Blank checks and pure-element check samples
- Spectrace 9000 application software (this software, which is device-specific and not interchangeable, is identified by the serial number of the unit)
- XRF sample cups
- Battery charger
- Instruction manual
- Shipping/carrying case

8.0 PROCEDURE

The **operator** shall perform the following procedure, making any deviations from this SOP in accordance with QP-5.7 and/or SOP-01.01.

8.1 Perform Initial Setup

- 8.1.1 Connect the battery.
- 8.1.2 Turn the unit on.
- 8.1.3 Set the time and date. (It is critical that the date is accurate because the source-decay compensation is based on the date.)
- 8.1.4 Allow the Spectrace 9000 to warm up for approximately 30 minutes.
- 8.1.5 Set desired application (soil, thin filter, lead in paint, etc.).
- 8.1.6 Set measuring times for radioactive sources.

Note: Measuring times depend upon the elements of interest as well as the desired detection limits. Minimum measuring time for each source should be 20 seconds. Although counting statistics

improve as measurement time increases, the practical limit for typical applications is 600 to 800 seconds. Refer to Attachment A for the manufacturer's guidance regarding elements excited by radioactive sources and detection limits.

- 8.1.7 Set time as "real" or "live."
- 8.1.8 Disable the display threshold.
- 8.1.9 Set instrument to store automatically the results and spectra (optional).

8.2 Use Probe

- 8.2.1 Make sure the probe is always in contact with the surface of the analyzed material and that the material completely covers the probe opening (aperture) when the sources are exposed.

- 8.2.2 Use the probe's laboratory, safety-shield assembly when the probe is inverted for measuring samples contained in cups.

Note: Do not remove a sample or move the probe while the screen displays the message "Source On" or while the base of the probe flashes.

- 8.2.3 During operation, keep the probe at least three feet from computer monitors or any other source of radio frequency that could affect measurement results.

8.3 Perform Preoperational Checks

- 8.3.1 Analyze the following preoperational-check samples prior to sample analysis:

- Energy calibration check
- Resolution check
- Blank sample check
- Target metals response check

Note: Refer to the Spectrace 9000 training manual and to Spectrace's "Standard Operating Check," August 15, 1994, for detailed procedures on preoperational checks.

- 8.3.2 Once at the beginning of each day, perform element x-ray response and spectrum resolution check using the Fe blank, recording results in the field or instrument logbook.

- 8.3.3 If intensities are outside the specified range, refer to Spectrace's "Standard Operating Check" and perform an energy calibration.

- 8.3.4 If the Kev value noted in the spectra of the energy calibration check is out of range, refer to the “Standard Operating Check” and perform an energy calibration.
- 8.3.5 Perform analytical background check with the Teflon blank at least once a day, recording results in the field or instrument logbook.
- 8.3.6 If the element concentrations of the elements of concern are greater than five times the standard deviation, refer to the “Standard Operating Check”; may perform the Acquire Background procedure.
- 8.3.7 Analyze two standard-reference materials (SRMs) (e.g., National Institute of Standards and Technology [NIST] SRM 2710 or 2711) or performance-evaluation samples with metal concentrations; bracket the known or suspected metal concentrations of the measured samples.
- Note:** If using the instrument for quantitative work, perform this check at least once a day and record results in the field or instrument logbook to note significant trends in the analytical measurement.
- 8.3.8 Determine the precision by making multiple analyses of either an SRM (NIST SRM 2710 or 2711) or a thoroughly characterized sample for an evaluation of the precision of the elements of interest.
- Note:** If using the instrument for quantitative work, perform this at least once a day or once every 20 samples. Conduct this measurement under the same conditions as those used for sample measurements. Record results in the field or instrument logbook. The precision objective should be a 20% relative-standard deviation.

8.4 Handle and Present Samples

The following procedures are recommended for handling and preparing the samples.

8.4.1 Soil Samples Application

Analyze soil samples in either in-situ or in bench-top mode.

8.4.1.1 In-Situ Measurement

- Prepare soil by stirring it with a trowel (or similar utensil).

- Remove rocks, vegetation, or large objects; may sieve soils.
- Flatten soil with a trowel (or similar utensil).
- Place the probe securely against the flattened soil surface.
- Secure the probe to ensure that it does not move or topple over.
- Push either the trigger button on the probe or the “CONT” button on the instrument to initiate the measurement.

8.4.1.2 Bench-Top Measurement

- Ensure that the sample moisture content is less than that at saturation.
- Prepare the soil until obtain the desired homogeneity.
- Put the soil into a sample cup and seal it with x-ray film.
- Push the “CONT” button on the instrument to initiate the measurement.

8.4.2 Thin (Filter) Samples Application

- 8.4.2.1 Mount the filter and place on the probe, ensuring that the filters are presented loaded-side down and wrinkle-free.
- 8.4.2.2 Use the probe safety cover when measuring thin samples.
- 8.4.2.3 Visually prescreen filters and wipes for defects before using them to establish background and contamination levels.

8.4.3 Lead-in-Paint Application

- 8.4.3.1 Check that the area selected for analysis is smooth, representative, and free of surface dirt.
- 8.4.3.2 Hold the Spectrace 9000 probe firmly on the surface.

8.4.3.3 When using the instrument for specimen applications (e.g., on paint chips or nonbacked films), use the probe's safety cover.

8.5 Perform Data Quality Assurance/Quality Control

Run a certified-reference sample periodically. This provides valuable accuracy and precision data. Running a certified-reference sample also alerts the operator that either the probe window is contaminated or the instrument is not operating properly.

8.6 Record and Document Results

8.6.1 Store results (and spectra) automatically; refer to the manufacturer's operating manual.

8.6.2 Download results to a personal computer at the end of each day; refer to the Spectrace 9000 training and applications manual.

8.6.3 Transfer results to a spreadsheet.

8.6.4 If qualitative data (i.e., used only for general surveys or to bias sampling), handle data informally and record in the field or instrument logbook.

8.6.5 The **data technician** shall transfer RRES-RS project decision data to the Environmental Restoration Database (ERDB) following a peer review.

8.6.6 Report results for a blank and a check standard.

9.0 LESSONS LEARNED

9.1 Before performing work described in this SOP, **RRES-RS project participants** should go to the Department of Energy Lessons Learned Information Services home page, located at <http://www.tis.eh.doe.gov/II/II.html>, and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.

9.2 During work performance and/or after the completion of work activities, **RRES-RS project participants**, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

10.0 RECORDS

The **field team leader** shall submit the following records to the Records Processing Facility, in accordance with QP-4.4:

- Completed daily activity log or instrument log book, which should include the following pieces of information:
 - Record that the leak test has been performed
 - Element X-ray response check
 - Analytical background check
 - Standards check
 - Energy calibration
 - Performance evaluation
 - Sample preparation
 - Measurement time for each radioactive source
 - Sample results
 - Precision results

11.0 REFERENCES

To properly implement this SOP, **RRES-RS project participants** should become familiar with the contents of the following documents located at http://erinternal.lanl.gov/home_links/Library_proc.shtml:

- RRES-RS Quality Management Plan
- QP-2.2, Personnel Orientation and Training
- QP-4.4, Record Transmittal to the Records Processing Facility
- QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities
- SOP-01.04, Sample Control and Field Documentation
- EPA (U.S. Environmental Protection Agency), "Representative Sampling Guidance, Volume 1—Soil," (OSWER Directive 9360.4-10) (EPA Environmental Response Team, Washington, DC, November 1991).
- EPA (U.S. Environmental Protection Agency), "Spectrace 9000 Field Portable X-Ray Fluorescence Operating Procedure" (SOP 1713) (EPA Environmental Response Team, Response Engineering and Analytical Contract Standard Operating Procedures, Washington, DC, December 21, 1992).
- NIST (National Institute of Standards and Technology), "Standard Reference Material (SRM) 2710, Montana I Soil, Highly Elevated Trace Elements Concentrations, Certification of Analysis," (Standard Reference Materials Program, NIST, Gaithersburg, MD, October 30, 1992).

- NIST (National Institute of Standards and Technology), "Standard Reference Material (SRM) 2711, Montana II Soil, Moderately Elevated Trace Elements Concentrations, Certification of Analysis," (Standard Reference Materials Program, NIST, Gaithersburg, MD, October 30, 1992).
- TN Technologies, Inc. (now TN Spectrace), "Spectrace 9000 Portable XRF Analyzer Manufacturers Operator Manual, Part No. 717715, Rev. II," (TN Technologies, Inc., August 1994).
- TN Technologies, Inc. (now TN Spectrace), "Spectrace 9000 Standard Operating Check," (TN Technologies, Inc., August 15, 1994).
- TN Technologies, Inc. (now TN Spectrace), "Spectrace 9000 Field-Portable/Benchtop XRF Training and Applications Manual," (TN Technologies, Inc., 1994).

Note: TN Spectrace is located at 2555 North Interstate Highway 35, P.O. Box 800, Round Rock, Texas 78680-0800; (800) 736-0801]

12.0 ATTACHMENTS

Attachment A: Elements Excited by Radioactive Sources and Lower Limits of Detection for These Elements (2 pages)

Attachment B: List of Acronyms and Abbreviations, (1 page)

[Using a token card, click here to record "self-study" training to this procedure.](#)

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

Attachment A: Elements Excited by Radioactive Sources and Lower Limits of Detection for These Elements

For soil samples application using source-measuring times of 60 s and 200 s, the following are typical element minimum detection levels (MDLs):

Source	Element	60s	200s
		MDL (mg/kg)	MDL (mg/kg)
⁵⁵ Fe	Potassium (K)	325	140
	Calcium (Ca)	150	75
	Titanium (Ti)	110	65
	Chromium (CrLo)	180	117
¹⁰⁹ Cd	Chromium (CrHi)	525	345
	Manganese (Mn)	410	320
	Iron (Fe)	225	155
	Cobalt (Co)	205	138
	Nickel (Ni)	125	95
	Copper (Cu)	90	55
	Zinc (Zn)	70	50
	Mercury (Hg)	60	50
	Arsenic (As)	50	35
	Selenium (Se)	35	25
	Lead (Pb)	30	20
	Rubidium (Rb)	10	10
	Strontium (Sr)	10	5
	Zirconium (Zr)	10	3
	Molybdenum (Mo)	10	4
²⁴¹ Am	Cadmium (Cd)	180	90
	Tin (Sn)	100	50
	Antimony (Sb)	65	35
	Barium (Ba)	20	10
	Uranium (U)	100	60
	Thorium (Th)	100	60
	Silver (Ag)	100	60

For thin-sample application using a source measuring time of 200s for the ^{55}Fe and ^{109}Cd sources, and 800s for the ^{241}Am source, the following are typical element MDLs:

Source	Element	MDL (g/cm ²)
^{55}Fe	Potassium (K)	0.40
	Calcium (Ca)	0.20
	Titanium (Ti)	0.15
	Chromium (CrLo)	0.40
^{109}Cd	Chromium (CrHi)	0.90
	Manganese (Mn)	0.65
	Iron (Fe)	0.65
	Cobalt (Co)	0.50
	Nickel (Ni)	0.30
	Copper (Cu)	0.65
	Zinc (Zn)	0.40
	Mercury (Hg)	0.45
	Arsenic (As)	0.40
	Selenium (Se)	0.15
	Lead (Pb)	0.50
	Rubidium (Rb)	0.10
	Strontium (Sr)	0.10
	Zirconium (Zr)	0.15
	Molybdenum (Mo)	0.10
^{241}Am	Cadmium (Cd)	2.5
	Tin (Sn)	2.5
	Antimony (Sb)	1.5
	Barium (Ba)	0.70

Attachment B: List of Acronyms and Abbreviations

ERDB	environmental restoration database
MDL	minimum detection level
LANL	Los Alamos National Laboratory
NIST	National Institute of Standards and Technology
PPE	personal protective equipment
PTL	project team leader
QII	quality integration and improvement
QP	quality procedure
QPPL	quality program project leader
RRES-RS	risk reduction and environmental stewardship—remediation services
SOP	standard operating procedure
SRM	standard reference material
SSHASP	site-specific health and safety plan